Design of Cobalt ferrite-doped bismuth ferrite based composites for multiferroic application

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

by

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It is the work that is expected to be carried out under the supervision of Dr. Jai Parkash and co-supervision of Dr. Jitendra Pal Singh.

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ABSTRACT

The research is devoted to employing the solid-state reaction method to synthesize multiferroic application composites of Bismuth ferrite ($BiFeO_3$) and Cobalt ferrite ($CoFe_2O_4$). The structural behavior of cobalt ferrite and doped bismuth ferrite will be analyzed using powder x-ray diffraction methods (XRD) and data will be analyzed using Fullprof software. The dielectric behavior will be determined using LCR meter (inductance, resistance, and capacitance meter). Parameters that include loss tangent, dielectric loss, and dielectric constant will be studied at different selected frequencies. The Vibrating Sample Magnetometer (VSM measures the magnetic characteristics, which will used to calculate the coercive field and magnetization, which can be attained from hysteresis loops. The objective of our work will be to improve the dielectric and magnetic properties apart from previous literature successes.

Key words: Multiferroic – composites, Cobalt ferrite (CFO), Bismuth ferrite (BFO), XRD, LCR.

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1. Introduction

In the past few years, multiferroic materials have garnered attention for their unique "ferroic" features, leading to significant research and development for many applications [1,2]. Many forms of multi-ferroics are created on their magneto-electric coupling, lone-pair activeness, magnetically driven ferro-electricity, geometric ferro-electricity, charge ordering, and multi-ferroic composites. Thus, CFO-BFO composites displays higher magneto-electric properties rather than other type of multi-ferroic composites. Thus, the multiferroic composite combines with two materials with substantial magnetic and electrical properties, which results in strong magneto-electric interaction [3]. There are numerous materials combinations which exhibits distinct magneto-electric coupling mechanisms while includes strain and charge [4-7]. Hence, CFO possess a high curie temperature of almost 485^0C, while BFO which is an anti-ferromagnetic material possess a high neèl temperature of about 640^0C. BFO is well known single phase multiferroic material at a room temperature (RT) with electric polarization from the lone pair of Bi^{3+} (2s orbital) and Fe^{3+} play an important role for displaying the magnetization in the system. $BiFeO_3$ shows antiferromagnetic at RT, along with G – type ordering is due the half-filled or partially filled d-orbital of Fe^{3+} ion. [3,8]. CFO-BXFO composites may demonstrates enhanced magnetization and a preeminent dielectric constant. Thus, the BFO-CFO composite is a well-known multi-ferroic composite that has undergone extensive development. Hence, BFO-CFO nano-composites exhibits exceptional magneto-electric coupling and unique properties [9-11]. Thus, CFO is a ferro-magnetic materials made up of Fe^{3+} and Co^{2+} ions, and possess high magnetostriction compared to other rare-earth ferro-magnetic materials [12]. The ferro-electric leads to possess BFO and ferro-magnetic characteristics of CFO plays a dynamic role in achieving a significant magneto-electric coupling [8]. Thus, combining BFO and CFO results in anti-ferromagnetic-ferromagnetic exchange couplings and improved magnetic properties [13-15]. Since, BFO-CFO has the

unique features of photoactivity and stability in alkaline environments makes it more suitable for future photo-voltaics, photo-catalytic, and electro-catalytic devices.

1.1 Types of ferrites

Ferrites are categorized into two categories depending upon their magnetic coercivity and the resistance to demagnetize that is soft and hard ferrites [16].

1.1.1 Soft Ferrite

Soft ferrites are electromagnetic materials which have certain properties such as: hardness and brittleness. Soft ferrites shows lower magnetism, even after magnetization is supposed to be permanent within hard ferrites. The term "Soft Ferrite" tends to a group of ceramic and electro - magnetic materials. Soft ferrites materials are class of inverse spinel structure, which is made up of divalent metal ion and transition metal ion. However, according to magnetic domain theory (MDT), shows that the formation of small magnetized regions within the material, which is known as magnetic domains [17]. Consequently, the net magnetic flux in the system is zero. First class of ferrites (Soft ferrites) display low coercivity, low retentivity, and a fine hysteresis curve [18].

1.1.2 Hard Ferrite

Hard ferrites retain a permanent magnetic moment even when there is no magnetic field present. Hard ferrites are permanent and magnetic in nature [19]. They have high coercivity high retentivity and a large area of hysteresis curve.

$CoFe_2O_4$ structure using VESTA.

$CoFe_2O_4$ has an inverse spinel structure in the cubic unit shell. $F\bar{d}3m$ has a space group number of 227. FeO_6 octahedra are formed by attaching Fe^{3+} ions to six equivalent O^{2-} atoms. These octahedra share corners with CoO_4 tetrahedral and edges with FeO_6 octahedra. All Fe-O bond lengths are 2.04 Å. Co^{2+} ions bond with four equivalent O^{2-} ions to produce CoO_4

tetrahedra, which share corners with twelve equivalent FeO_6 octahedra. The corner-sharing octahedral angles are tilted by 58°.

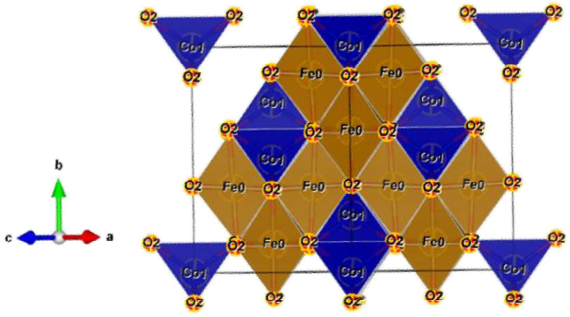

Fig.: Structure of CFO

All Co-O bond lengths are 1.96 Å. OFe_3Co trigonal pyramids are formed by bonding one O^{2-} ion to three equivalent Fe^{3+} and one Co^{2+} ions, resulting in deformed edges and shared corners. [20]

$BiFeO_3$ structure using VESTA.

$BiFeO_3$ has a trigonal structure that belongs to the R3c space group. Fe^{3+} is attached to six equivalent O^{2-} atoms, developing distorted corner-sharing FeO_6 octahedra. The corner-sharing octahedral tilt angles are 29 degrees. The Fe-O bond lengths range from 1.95 Å to 2.16 Å. Bi^{3+} is bound with six-coordinate geometry along with 6 equivalent O^{2-} atoms.

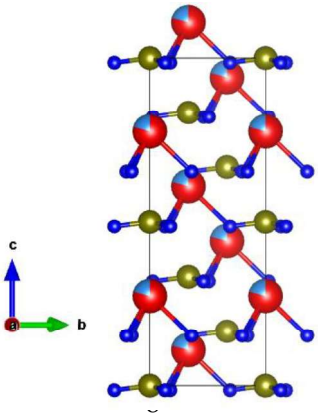

Fig.: Structure of BFO

2. Literature Review

Yadav et.al. synthesized a composite of $(x)Co_{0.5}Ni_{0.5}Fe_2O_4$-$(1-x)Bi_{0.9}La_{0.1}FeO_3$ using the solid-state reaction method. The XRD pattern of nickel-doped cobalt ferrite (CNFO) and lanthanum-doped bismuth ferrite (BLFO) confirms that impure phase is present in composites. The value of the dielectric constant measured at two different frequencies (500 kHz and 1 MHz), increases on increasing the ferrite concentration. Further, the magnetic property increases on increasing the ferrite concentration. Ferroelectric measurement performed at room temperature. Ferroelectric ordering is indicated by the polarization (P) - electric (E) hysteresis loops at room temperature, and the magnetodielectric effect at room temperature confirms the link between ferroelectric and ferromagnetic orderings [21].

Das et.al synthesized composites having the chemical formula $x\ CoFe_2O_4$ - $(1-x)BiFeO_3$ (x = 0.2, 0.3, and 0.4) using the sol-gel method. According to XRD analysis the presence of CFO and BFO phases in composites were identified, with minor impurities. At room temperature hysteresis loops of composites exhibits a substantial increase in magnetization values when it is compared with pure BFO and observed increasing with CFO content. The frequency-dependent di-electric constant (ε_r) curves exhibits dispersive behaviour in low-frequency range which demonstrates the existence of relaxation along the grain boundaries. The ε_r-T charts for all materials exhibits a gradual increase in ε_r with increase in temperature, with an alteration near at 700 K, which solely corresponds towards the anti-ferromagnetic néel temperature of BFO [22].

Dabas et.al synthesized CFO-BFO composites using a conventional solid-state reaction method. The X – ray Diffraction analysis reveals that there is a presence of impure phase in CFO–BFO composites. It was identified that the ferro-electric polarization of composite

samples shows reduction in values which are followed by decrease in dielectric loss due to in-corporation of magnetic phase (CFO) into composites. Composites having a minimal loss, makes them appropriate for spintronics applications and memory storage devices [23].

Khalid et.al synthesized (1-x)$BiFe_{0.95}Mn_{0.05}O_3$-x $CoFe_2O_4$ composites samples by using the Sol-gel auto combustion method with x values of 0.2, 0.3, 0.4, 0.5, 0.6, and 0.7. XRD spectra indicated the presence of both phases (perovskite and spinel) of composite. On increasing the CFO concentration, the synthesized series exhibits consistent dielectric properties. Ferro-electric analysis of coreshells nanocomposites shows that there is a substantial increase in ferro-electric appearance with CFO concentrations. The magnetic study reveals that a significant increase in saturate magnetization (7.79-26.41 emu/g) and remanence magnetization (3.23-1.27 emu/g), while coercivity remains consistent among all the composites. Throughout all the samples it possess a significant magneto-electric (ME) coupling, which possess potential for the application of storage devices [24].

Sharma et al. synthesized composites of BFO and CFO using sol-gel method. XRD data analysis shows the presence of both phases. The composites 0.5BFO–0.5CFO and 0.3BFO–0.7CFO shows high dielectric constants. BFO demonstrates weak ferromagnetism, having 374 Oe coercivity (H_c) and 6×10^{-2} magnetisation (M_s) at 21 kOe. The composite samples exhibit ferrimagnetic properties and shows decrease in remanence magnetization and saturation when the BFO content increases. However, coercivity differs with BFO level, 0.8BFO-0.2CFO exhibiting the highest H_c [25].

Basov et.al offered a new and effective technique for creating multiferroic nanocomposites that are vertically aligned. The technique enabled the electrodeposition of $CoFe_2$ nanopillar arrays through an anodic aluminium oxide template supported on a Si substrate, resulting in regular, dense, and free-standing arrays. Subsequently, a ferroelectric film was directly radio frequency (rf) sputter-deposited onto the pillar structure to form $Ba_{0.70}Sr_{0.30}TiO_3$-$CoFe_2O_4$ multiferroic nanostructures. This was confirmed by both structural and magnetic characterization, resulting in in-situ oxidation of the ferromagnetic nanopillars [26].

Muneeswaran et.al synthesized the composite of (1−x) BFO- x CFO (CFO, x=0.00, 0.05, 0.10, and 0.30). Composition-driven structural transition from trigonal (R3c) to tetragonal (P4mm) is confirmed by Rietveld refinement analysis based on XRD data. Raman spectra revealed two phonon-scattering modes at higher wavenumbers, supporting the structural transition of the BFO-CFO crystal structure [27].

Younas et.al synthesized spinel-perovskite-based nanocomposites (x) CoFe2O4-(1-x) BiFeO3 (x = 0.0, 0.1, 0.2, and 0.3) created using a two-step process. Discrete $BiFeO_3$ and $CoFe_2O_4$ were first combusted using sol-gel auto-combustion, then mixed using an agate mortar pestle. Structural investigations found a rhombohedral phase of BFO and inverse spinel of CFO, indicating the development of composites. The dielectric properties showed a significant decrease in tangent loss at x is 3. The dielectric constant value was compared with the value reported in the literature and found it to be high. These findings are promising for developing energy storage and miniaturized electrical gadgets [28].

Arifiadi et.al synthesized high-purity CFO-BFO nano-composites using sol-gel method. Magnetic characterizations show that nanocomposites exhibit greater coercivities than single-phase CFO, demonstrating the dipolar interaction between ferrimagnetic nanocomposites' CFO and BFO. At 20 kOe, magnetization values vary from 22 to 41 emu/g. According to P-E loops, nanocomposites exhibit leaky behaviour and have an energy storage capacity of 0.09–0.11 mJ/cm^3. The change in polarization was predicted around 4.0-295 $\mu C/m^2$ at 30 kOe magnetic field [29].

Xian-Ming Liu et.al synthesized unique $xCoFe_2O_4$-(1-x) $BiFeO_3$ spinel-perovskite nanocomposites with a composition of x = 0.1, 0.2, 0.3, and 0.4, made by thermal disintegration of the polymeric gel technique at temperatures between 400 and 800^0C. The crystallite size differs between 20 and 80 nm. The study examined how annealing temperature and $CoFe_2O_4$ concentration in composites affect the magnetic characteristics of the spinel-perovskite system. Annealing magnetoelectric composites at 700^0C results in excellent magnetic characteristics such as saturation magnetisation and coercivity. Increased

$CoFe_2O_4$ content in nanocomposites leads to higher saturation magnetisation and coercivity levels [30].

3. Description of broad area

The concurrent existence of ferro-electrics and ferro-magnetics properties in multiferroic materials has drawn a lot of attention these days. Due to multiple applications of these materials are used in multi-functional devices such actuators, memory chips, sensors, and energy storage devices. Due to the exceptional magnetic and electric properties, cobalt ferrite ($CoFe_2O_4$) and bismuth ferrite ($BiFeO_3$) respectively composites of these materials are the better option to prefer memory devices. The CFO-BFO based composites may exhibits ferromagnetic and ferroelectric. Bismuth ferrite is found to be a ferroelectric material exhibiting high Curie temperature and anti-ferromagnetic nature, while cobalt ferrite is known to be a ferrimagnetic material with strong coercivity and magneto-striction. The objective of this work is to utilize the solid-state method, which is scalable and cheaper way to create $CoFe_2O_4$-$BiFeO_3$ composites. To produce high-quality $CoFe_2O_4$-$BiFeO_3$ composites that may show both ferro-magnetic and ferro-electric characteristics using appropriate and effective process. Hence, to optimize the potential applications of these composites in next-generation multi-functional devices and for our better understanding of this material properties.

4. Objectives of this study

These are following research goals:

- Synthesis of $CoFe_2O_4$-$BiXFeO_3$ (X =Alkaline earth elements) material by using solid state reaction.
- Structural and morphology studies by X-ray diffraction (XRD) and Scanning electron microscopy (SEM) respectively.
- To study Magnetic properties of multiferroic material by using Vibrational Sample Magnetometer (VSM) / Superconducting quantum interference device (SQUID)

- To study dielectric properties of CFO-BXFO.

5. Methodology

5.1 Synthesis of Cobalt Ferrite ($CoFe_2O_4$)

CFO will be synthesised utilising using conventional Solid-State Method. Ferric oxide and cobalt oxide will be combined in proper stoichiometric composition. The powder of CoO and Fe_2O_3 were mixed for minimum 2 hours and calcined at 600°C for 10 hours to breakdown carbonates and higher oxides. The calcined powder will be blended for 2 hours, pressed into pellets, and sintered at 1000°C for 50 hours to yield bulk CFO.

5.2 Synthesis of BXFO (X = Mg, Ca, Sr and Ba)

BXFO will be synthesised using rapid two-stage reaction method. Bi_2O_3, Fe_2O_3, and XCO_3 (X = Mg, Ca, Sr and Ba) were taken in stoichiometric ratios having purity (99.9%). The raw material mixed using agate mortar for 2 hours. After that the fine powders were calcinated at different temperature depending on the dopant raw material. The calcinated powders were mixed again for 2 hours, fine powder filled into a dye and pressed upto 4 Ton pressure to and wait for 3 minutes. The pellet was sintered in a programable furnace at certain temperature and quenched the pellet.

5.3 Synthesis of CFO-BXFO Composites:

CFO-BXFO powder will be mixed for 2 hours in agate mortar pestle. The mixed powder was pressed into a pellet. The pellet was sintered at 800^0C for 10 min.

6. Characterization and Analysis

Nowadays, researchers are synthesising a novel material utilising various techniques such as sol-gel, solid-state reaction etc. We are approaching solid state reaction method for the synthesis of our material. These synthesized samples can be analysed to determine various physical properties like crystal structure, morphology, magnetic and dielectric. Characterisation of the substance using the following instruments:

- ❖ Morphology by using SEM.

- ❖ X-ray diffractometer will be used for structural properties.
- ❖ Dielectric constant is determined by using Inductance Capacitance Resistance (LCR) meter.
- ❖ Vibrational Sample Magnetometer (VSM) is utilised to measure the magnetic properties of materials.

7. Expected outcomes of the research

- It is expected that CFO-BXFO composites will depict rich magnetic and dielectric properties.
- The composites may show low value of loss tangent.

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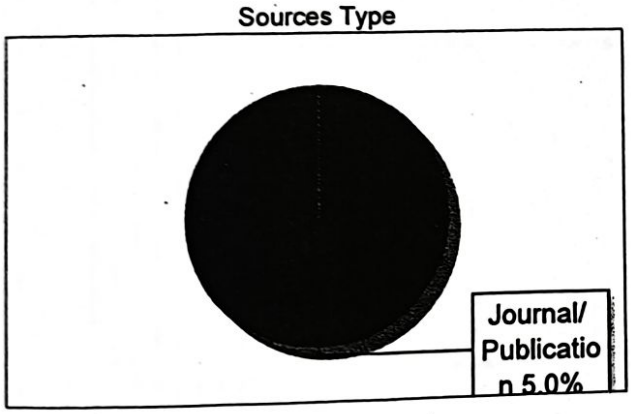

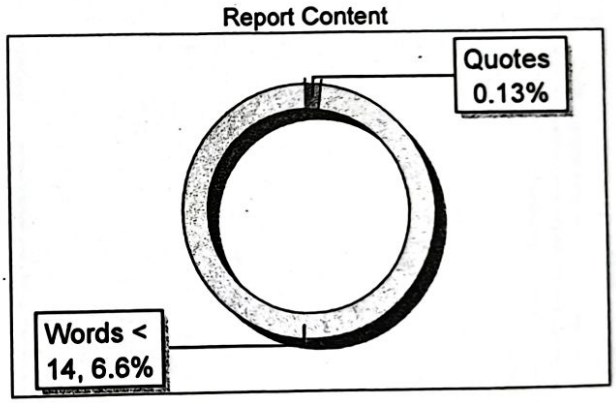

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Design of Cobalt ferrite-doped bismuth ferrite based composites for multiferroic application

SHORT SYNOPSIS
by

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2024

ABSTRACT

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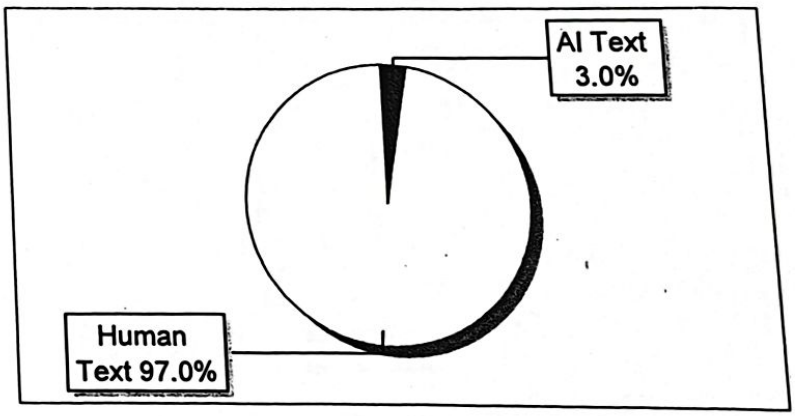

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